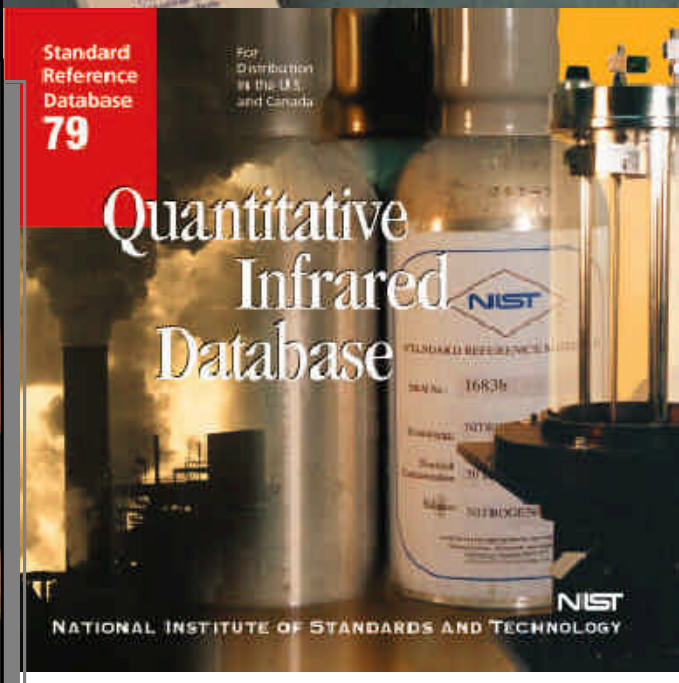

Protecting the Environment

Chemical Measurement Technologies

- Improving methods for certification of Hg and Sn in environmental SRMs
- Developing method with pyrohydrolysis extraction and ID-TIMS to quantify chlorine in complex matrices (to address chlorine emissions from fossil fuel burning)
- Working with the Defense Special Weapons Agency and the Organization for the Prohibition of Chemical Weapons to develop and critically evaluate chemical measurement protocols for use during onsite inspection of manufacturing and storage facilities
- Developing/implementing new efforts in using high resolution mass spectrometry for environmental applications
- Established protocols for assessing microhomogeneity of environmental SRMs; updating certificates of relevant SRMs
- Developing an infrared-based sensing device and evaluating a new microwave device for detection of oxygenated hydrocarbons in automobile exhaust
- Optimizing NIST-developed new measurement method for the determination of organic compounds (chloramines) that contribute to “residual chlorine” levels in sewage wastewater disinfected with chlorine
- Reduced oxygen interference in NIST ICP-MS determinations of sulfur by implementing the use of electrothermal vaporization for sample introduction
- Investigating new gas cylinder and preparation technologies to address challenges in producing gas mixture SRMs at lower concentration levels with lower total uncertainties as needed for emissions assessment by automobile manufacturers

Standards Development and Quality Assurance Activities:

- Developing ambient level gas standards for ambient global warming gases to support measurements by the atmospheric research community
- Continuing to partner with specialty gas companies to provide gas NIST Traceable Reference Materials (NTRMs) to support both Clean Air Act measurement needs and commerce
- Completed new nitric oxide and propane primary gas standards suites to support additional needed NTRMs types
- Issued NIST spectral reference database to support open-path Fourier transform infrared measurements of hazardous air pollutant (HAP) molecules
- Restocked all conductivity solution SRMs
- Developed Henry's Law Constants Database for environmentally significant compounds
- Developed and published PAH Structure Index in print and web formats
- Certified important analytes in several environmental matrix SRMs during the past year:
  - organic, inorganic, and organometallic constituents in air particulate, marine sediment and marine tissue matrices
  - trace elements in industrial sludge
  - lead in various household matrices
  - trace mercury in water and in coal
  - sulfur in various fuel-related materials
  - Used new NIST-developed methods based on multidimensional, class-separation approach to value-assign four tissue/sediment SRMs for toxic non-*ortho* PCBs
  - Certified suite of synthetic gasoline materials for benzene, total aromatics, total oxygen, sulfur content, and total olefin to provide NIST traceability for reformulated gasolines
- Established program and developing reference materials needed to support accreditation and oversight of private sector/state provision of proficiency test studies for EPA/States drinking and wastewater programs
- Provided measurement proficiency assessment for private laboratories and a wide range of other laboratories engaged in environmental assessment programs, such as the NOAA National Status & Trends Program
- Continued to establish analytical laboratory capabilities and marine environment specimen bank at NIST National Marine Analytical QA Program facility in Charleston, SC

New Infrared Database Introduced to Support Remote Sensing Measurements

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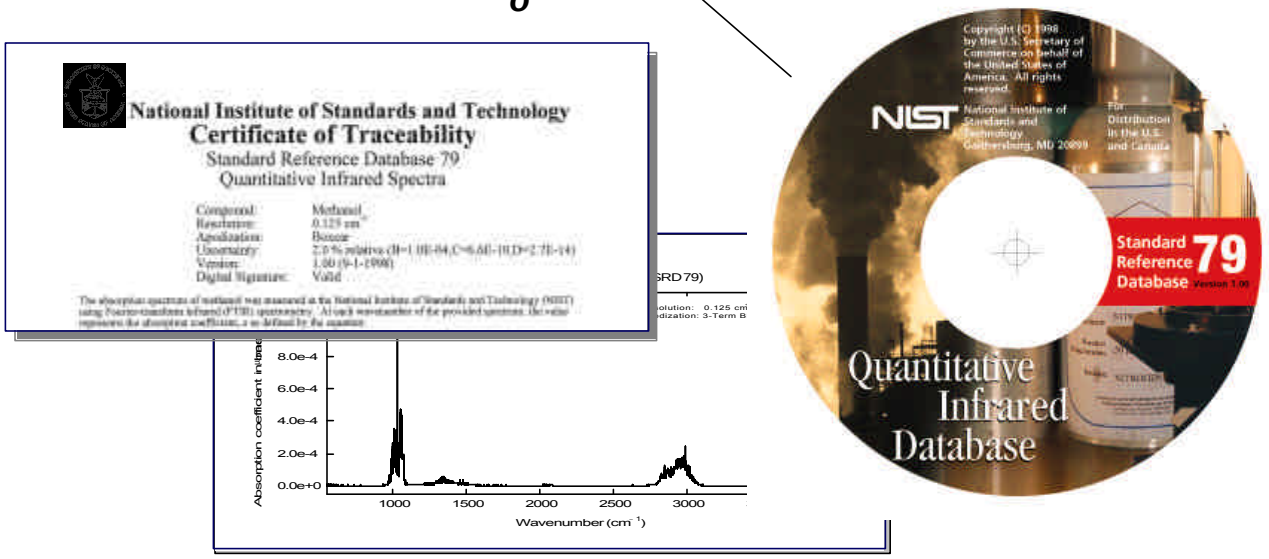


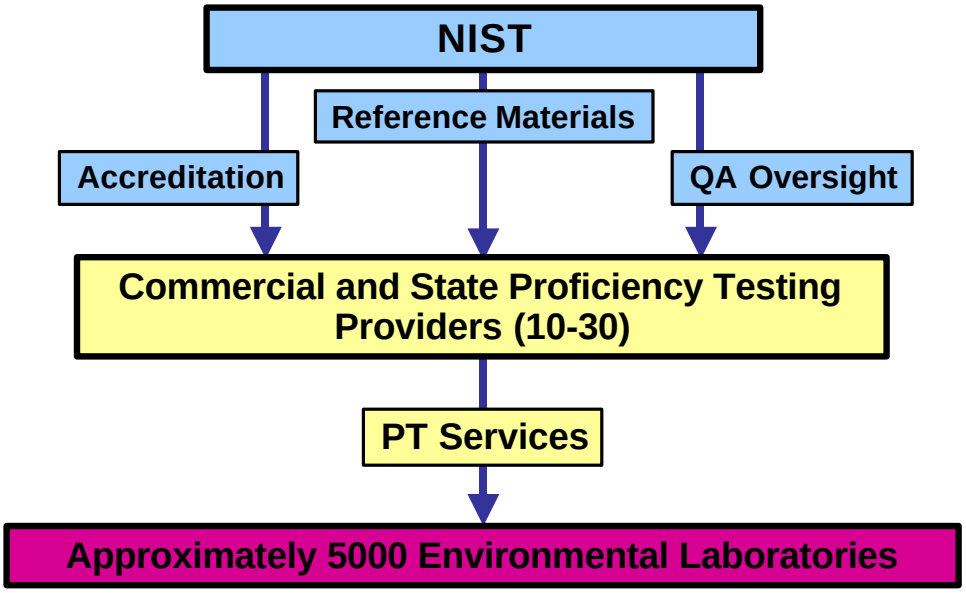
Project Description: Develop a quality-assured quantitative database of infrared spectra based on NIST gravimetric standards. The database will include absorption coefficients of approximately 100 of the 189 hazardous air pollutant molecules (HAPs) listed in the EPA's Clean Air Act Amendment of 1990.

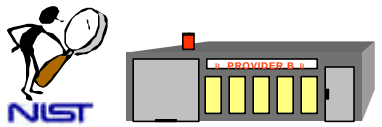
Results:

- **FY'00 Version 1.1 released**
  Absorption coefficient data for 9 additional compounds
  Update for 1 compound
- **FY '98 SRD 79 Version 1.0 released as CD-ROM**
  Absorption coefficient data for 21 compounds
  Traceable to NIST primary gas standards
  Comprehensive uncertainty analysis
  Digitally signed
  Internet updates

Relevance: Infrared based remote sensing technologies are being developed for real-time monitoring of airborne chemical emissions along plant boundaries and within plant facilities. This database is a critical part of the infrastructure required to establish infrared measurements as reliable and cost effective quantitative tools for environmental monitoring.

NIST Commences Accreditation/Oversight of Proficiency Test Study Providers for EPA/States Water Programs





Project Description: Working with the USEPA, States, and other public and commercial entities, NIST has established a system under which private-sector and interested states can be accredited by NIST to provide proficiency testing (PT) services to those laboratories testing drinking water and wastewater for regulated chemical, radiological, and microbiological parameters for USEPA and State water programs.

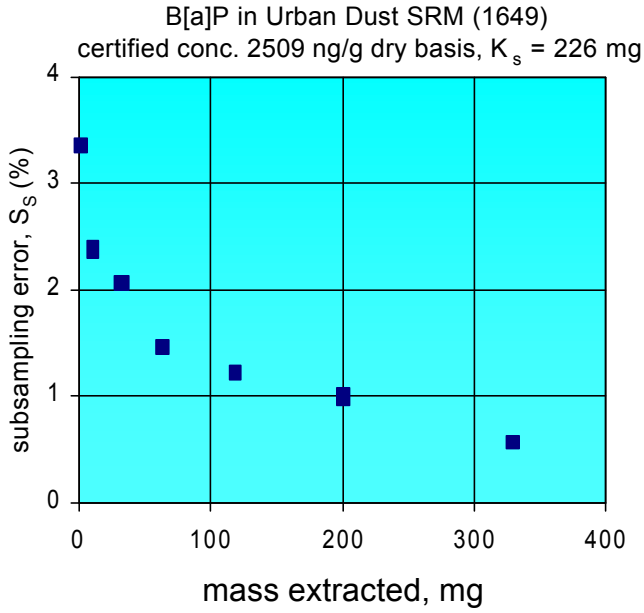
Relevance:

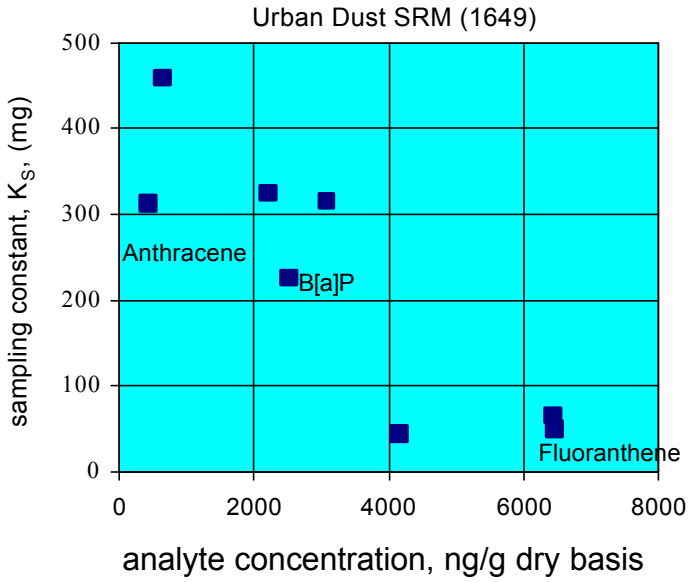
- EPA-conducted, semiannual testing to assess the competence of laboratories to conduct analyses for the Clean Water and the Safe Drinking Water Acts is being replaced by a multiprovider system in which NIST-accredited state and private-sector entities will provide these PT services on a fee-basis.
- Mechanism and tools to provide appropriate government oversight was critical in ensuring user acceptability of this new system.

Results:

- Issued Provider Technical Requirements: “NIST Handbook 150-19: Chemical Calibration -- Providers of Proficiency Testing”
- Accredited >12 Testing Providers in 48 program areas
- Establishing ACD infrastructure and producing primary benchmark materials where SRMs not available
  - to provide the infrastructure and primary standards needed to audit commercial PT samples
  - to assist providers in value-assigning their PT materials

Chemical Microhomogeneity of Natural Matrix Environmental SRMs for Organic Components





Relevance and Impact: As methods become more sensitive, analysts are using smaller sample sizes for analysis. SRM homogeneity needs to be demonstrated at these smaller mass levels.

Project Description: During the certification process of an SRM, particularly that of natural matrix materials, substantial effort is directed toward the determination of the homogeneity of the material with respect to a given sample size and a specific analyte. At present, the minimum sample size below which analyte homogeneity has not been assessed is stated in the Certificate of Analysis and typically ranges from several hundreds of milligrams to 10 g. The microhomogeneity (1 mg to 700 mg) of three SRMs: SRM 1649a, Urban Dust/Organics; SRM 1650a, Diesel Particulate Matter; and SRM 1944, New York/New Jersey Waterway Sediment, has been investigated for heterogeneity of selected polycyclic aromatic hydrocarbons (PAHs) in these materials.

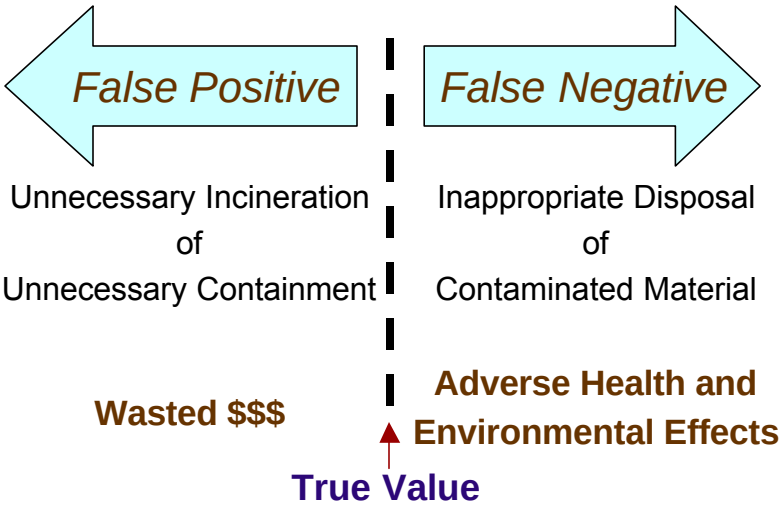
Results: Analytical and statistical procedures have been developed to assess the chemical microheterogeneity of these natural matrix SRMs for organic components. The Ingamells sampling constant (K_s), which is defined as the sample size of which the uncertainty due to sample heterogeneity is 1 %, has been used as the first approximation to the microhomogeneity of these materials. Results using this approach varied with the PAH concentration and ranged from 50 mg to 450 mg in the Urban Dust SRM and from 40 mg to 75 mg in the Diesel Particulate Matter SRM.

New Sediment SRM Enhances Accuracy of Environmental Measurements

SRM 1944, NY/NJ Waterway Sediment

- **SRM provides accuracy base for reliable decision-making**
- **Produced in collaboration with NY District Army Corps of Engineers and Environment Canada**
- **First NIST natural matrix SRM with values assigned for polychlorinated dibenzo-*p*-dioxins and dibenzofurans**
  - Certified values for >70 analytes
  - Reference values for >80 analytes

Impact of Analytical Accuracies on Environmental Decisions



Quantity of dredged material for disposal: > 4 x 10^6 m^3 per year

- **Disposal costs:**
  - For unrestricted disposal: **$30 M per year**
  - For containment: **$150 M to $600 M per year**
- **Testing costs per federal project: $1 M per year**

- U.S. Army Corps of Engineers - New York District

"Economic Impact of Standard Reference Materials for Sulfur in Fossil Fuels" - Report just released:

Assesses impact on industries including:

- the sulfur measurement industry
- the fossil fuels extraction and processing industry
- primary users of fossil fuels including the electric utility industry and steel industry

Measure	***Economic Impact***
Benefit-cost ratio	**113**
Social rate of return	**1,056%**
Net present value ($1998)	**$409,002,097**

Examples of New High-Priority SRMs Under Development

Environmental

- **Fish Fillet**
  (with *toxic contaminants such as PCBs, pesticides, and methylmercury along with food proximates to be certified)*

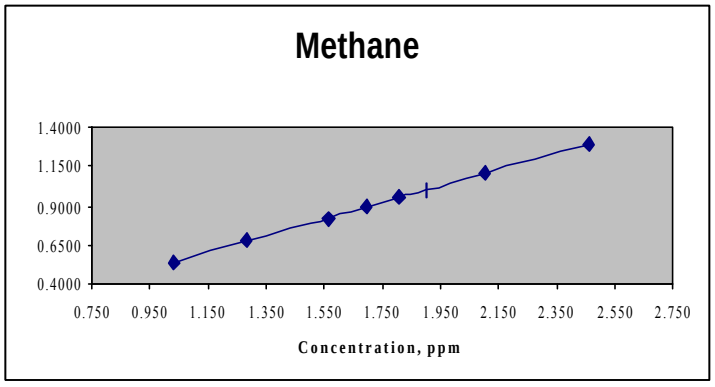
- **New Series of Low Concentration Nitric Oxide Gas Standards**
  (to support measurements needed for development of very low emission vehicles and future automobile emissions regulations)

  >45 Solution SRMs of Organic Analytes of Interest in Drinking Water and Wastewater Analysis, including:
  - **Volatile Organic Compounds in Methanol**
  - **Chlorinated Herbicides**
  - **Toxaphene in Methanol**
  - **Carbamates and Vydate in Acetonitrile**
  - **Haloacetic Acid Mixture in MTBE** (Water disinfection byproducts)

  (to provide accuracy benchmark and measurement traceability for proficiency tests provided to >5000 environmental laboratories by commercial vendors)

SRM®

Development of National Standards for Global Warming Gases



Project Description:

- trace gases in atmosphere are associated with climate change
- methane and nitrous oxide identified as greenhouse gases
- CFC-11 and CFC-12 rise to stratosphere and destroy ozone layer
- national gas standards needed to support treaty agreements
- develop set of national laboratory traceable gas standards to calibrate the measurement of selected ambient global warming gases

Results :

prepared set of 8 gas standards in an air matrix (that bracketed the nominal atmospheric concentrations)

methane (CH_4) : 1.033 - 2.457 ppm (± 0.2%)
nitrous oxide (N_2O) : 207.0 - 447.8 ppb (± 0.2%)
CFC-11 (CCl_3F) : 165.8 - 392.5 ppt (± 0.3%)
CFC-12 (CCl_2F_2) 276.2 - 555.3 ppt (± 0.3%)

- analytical agreement between NIST standards in set was 0.3 % or better
- sets compared with NOAA standards; as much as 7% differences

Relevance :

- need to track concentration of trace species in the atmosphere
- need to relate all monitoring data to national standards
- need to research standards development and come to closure between the NIST standards and those of NOAA, the US monitoring agency

Urban Air Particulate Matter on Filter SRMs

Fig. 1
Cyclone and filter assembly of air sampler

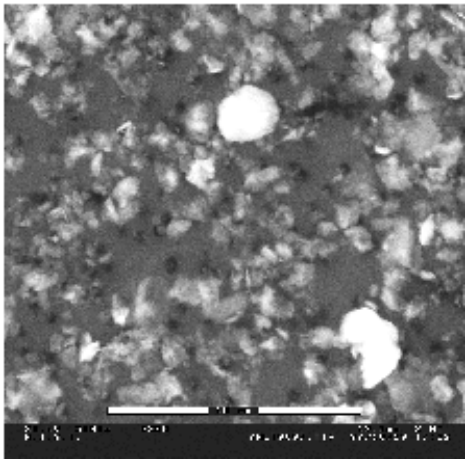


Fig. 2 Electron microprobe image

Project Description: To produce filter-based SRMs from contemporary fine particle $PM_{2.5}$ aerosol material (particulate matter smaller than 2.5 μm aerodynamic diameter – the high risk respirable fraction) to support EPA's national aerosol monitoring program.

Results: A high volume air sampling system shown in Fig. 1 was developed that separates fine particles with a cyclone separator and deposits them onto an array of filters. About 800 000 m^3 of air were processed from an urban area, collecting ~12 g of aerosol at a cut-off point of 1.7 μm which was used to produce material for SRM 2784 for the carbon analysis community. An electron microprobe image of a $PM_{2.5}$ aerosol on filters is shown in Fig. 2 (scale bar = 10 μm).

A $PM_{2.5}$ aerosol from Vienna, Austria was used to produce 2000 APM filters for SRM 2783, Urban Air Particulate Matter on Filter for Trace Elements. The SRM units were prepared from aqueous suspensions of the aerosol by filtering aliquots through 47 mm diameter polycarbonate filters with 0.4 μm pore size. Aerosol loadings on the prepared filters are 480 μg per filter on 10 cm^2. The homogeneity of the SRM lot has been confirmed by gravimetry and INAA. The concentration of >20 elements are being certified in this material.

Relevance: This unique production allows NIST to produce simulated APM filters. These key SRMs will be used by national and international aerosol monitoring programs and to support development of new EPA $PM_{2.5}$ air quality standards.

Ozone Standards Program

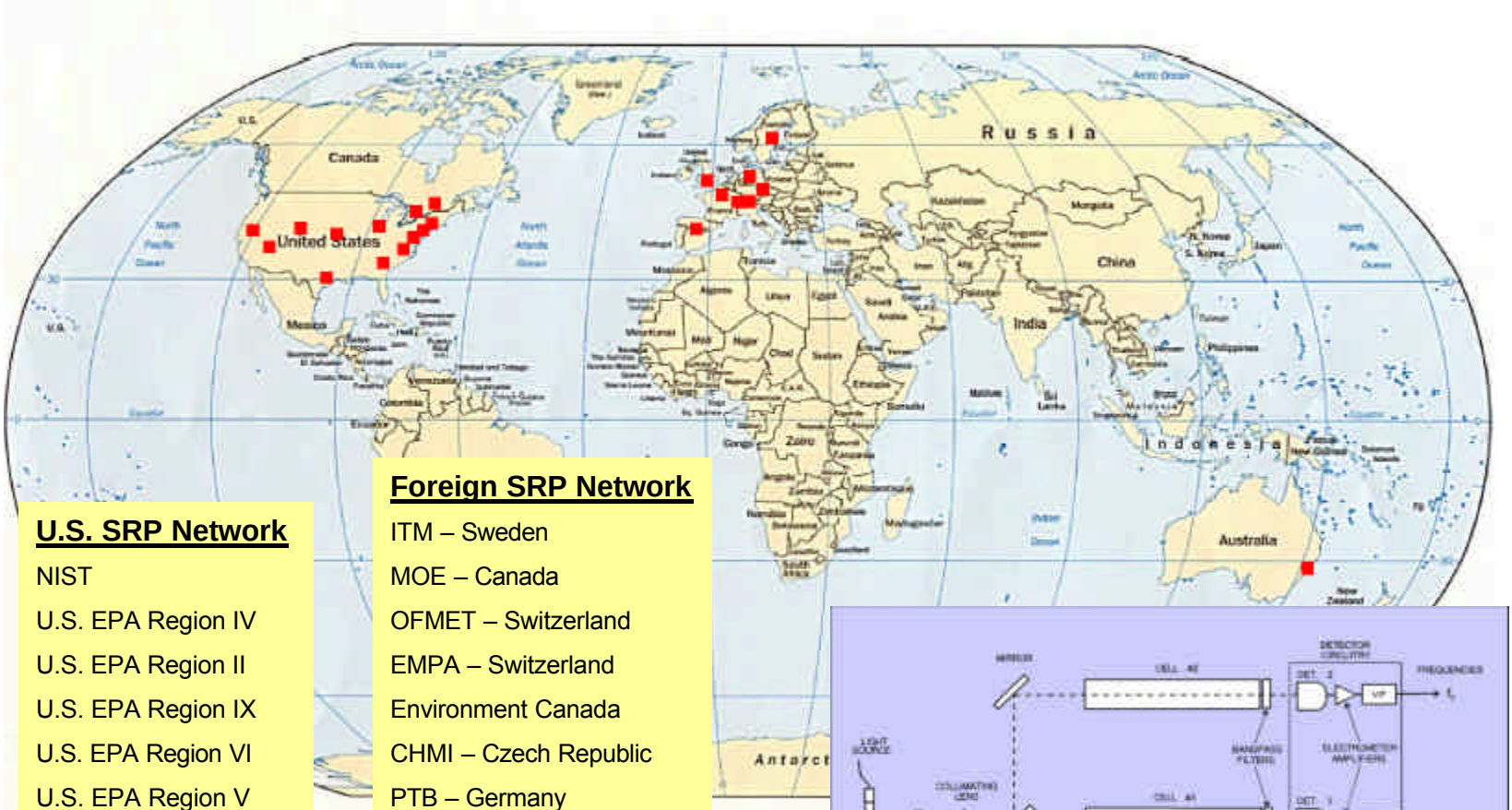
Project Description: To develop and maintain of a network of Standard Reference Photometers (SRP) certified against the U.S. National Reference Photometer maintained at NIST to provide ozone measurement traceability to NIST.

Results:

- Provided traceability to NIST for ozone measurements for:
  - U.S. ambient ozone measurements under U.S. EPA regulation
  - an increasing number of worldwide National Measurement Laboratories
- SRP traceability to NIST better than 1% agreement.
- Expanded Standard Uncertainty: 1 nmol/mol (0 – 100 nmol/mol) (absolute), 1% (100 – 1000 nmol/mol) (relative)

Relevance:

- Maintains mechanism for worldwide traceability of ozone measurements to NIST.
- Provides NIST traceability for U.S. Industry, Academia, and other Government Agencies.



U.S. SRP Network

NIST
U.S. EPA Region IV
U.S. EPA Region II
U.S. EPA Region IX
U.S. EPA Region VI
U.S. EPA Region V
U.S. EPA Region VIII
U.S. EPA Region I
U.S. EPA Region VII

Foreign SRP Network

ITM – Sweden
MOE – Canada
OFMET – Switzerland
EMPA – Switzerland
Environment Canada
CHMI – Czech Republic
PTB – Germany
NPL – United Kingdom
NSW-EPA – Australia
ISCIII – Spain
LNE - France